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By H. D. Jacobs and A. F. Colombo

UNITED STATES DEPARTMENT OF THE INTERIOR

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CATIONIC FLOTATION OF A HEMATITIC OXIDIZED TACONITE

by

H. D. Jacobs[1] and A. F. Colombo[2]

ABSTRACT

The Federal Bureau of Mines, as part of a program to insure future raw material needs for the iron and steel industry, undertook research to beneficiate the oxidized taconites from the western Mesabi range in Minnesota. The data presented were obtained by applying the Bureau-developed selective-flocculation/cationic-flotation process to the second of three oxidized taconite samples. These three samples are representative of an estimated 10 billion tons of ore analyzing 30 to 40 pct iron. The experimental work was performed in a 900-lb/hr pilot plant. Treating the ore sample (36.1 pct Fe) with the Bureau's process produced commercially acceptable concentrates averaging 63.5 pct Fe and 4.3 pct SiO_2, with an average iron recovery of 84.6 pct.

INTRODUCTION

To expand the U.S. iron reserve base, the Bureau of Mines has undertaken a research effort to develop beneficiation processes for the oxidized taconites[3] of the western Mesabi range. These reserves represent an estimated 10 billion tons (t)[4] of material analyzing 30 to 40 pct iron (7, 9).[5]

Three bulk samples (350 to 800 tons) of varying hematite-to-goethite ratio were obtained, representing three different types of oxidized taconites found on the Mesabi range. Since 1973, two processes--(1) selective flocculation and cationic flotation and (2) reduction roasting, magnetic separation, and cationic flotation--have been used to beneficiate the bulk samples. These processes are being investigated by the Bureau of Mines to decrease processing

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[3]The term "oxidized taconite" refers to material containing iron oxides that cannot be recovered by a drum-type magnetic separator with a maximum field strength of 3,000 gauss at its surface; the term is synonymous with "nonmagnetic taconite".
[4]Throughout this paper, ton (t) refers to a long ton or 2,240 lb.
[5]Underlined numbers in parentheses refer to the list of references at the end of this report.

costs and to establish a workable process for treating the different types of oxidized taconites encountered (1-4, 6, 8, 10). The first bulk sample was successfully treated by the selective flocculation and cationic flotation process and produced a concentrate containing about 62 pct Fe and 5 pct SiO_2 (6). This report covers the evaluation of the second bulk sample by the same process (5). Treatment of this sample of oxidized taconite second process, consisting of reduction roasting magnetic separation and cationic flotation, will be covered in a later report.

SAMPLE DESCRIPTION

The bulk sample consisted of 600 tons and was taken from a 9.5-mile-long block of oxidized taconite extending from approximately 5 miles west of Nashwauk to Coleraine, Minn. The sample represented about 1/2 to 1 billion tons of taconite. After crushing the oxidized taconite to minus 5/8 inch, a representative sample was taken and a size analysis was made (table 1). Mineralogically, the sample contained goethite, hematite, quartz, and trace amounts of magnetite, iron silicates, iron carbonates, and nonmetallic oxides. The hematite-to-goethite ratio was about 3 to 1. Liberation of the iron oxides from the gangue was at about 90 pct minus 44 μm (325 mesh). An average chemical analysis of the bulk sample is shown in table 2.

TABLE 1. - Screen analysis and iron distribution of oxidized taconite, pct[1]

Size fraction[2]	Weight	Iron	Iron distribution
Plus 1/2 inch	0.9	44.8	1.1
1/2 x 3/8 inch	2.0	43.8	2.4
3/8 x 1/4 inch	7.1	42.2	8.3
1/4 inch x 4 mesh	9.5	42.8	11.3
4 x 10 mesh	14.7	46.6	19.0
10 x 28 mesh	14.6	47.2	19.1
28 x 65 mesh	14.0	49.9	19.3
65 x 100 mesh	3.1	46.0	4.0
100 x 200 mesh	8.2	23.5	5.3
200 x 325 mesh	7.9	12.0	2.6
325 x 500 mesh	4.5	9.4	1.2
Minus 500 mesh	13.5	17.2	6.4
Composite	100.0	36.1	100.0

[1] Rodmill feed.
[2] Standard Tyler sieve sizes.

TABLE 2. - Chemical analysis of bulk sample, pct

Fe	36.1	CaO	0.41	Na_2O	0.05
Fe^{2+}	.5	S	.01	C	.11
SiO_2	44.5	P	.013	TiO_2	.26
Al_2O_3	.64	Mn	.08	LOI[1] at 400° C	1.6
MgO	.26	K_2O	.05	LOI[1] at 1,000° C	2.4

[1] LOI is loss on ignition.

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PILOT PLANT RESEARCH

Two flotation systems were extensively researched for upgrading the fine-grained, low-grade oxidized taconites found in the iron ranges of Michigan and Minnesota. One system employed anionic collectors and the other, cationic collectors. In both systems, the collectors selectively floated the siliceous gangue from the iron oxides. Bureau and industrial research has shown that the cationic system is the preferred process for treating oxidized taconites. This process comprises stage grinding of the taconite to liberation size to free the iron minerals from the gangue, selective flocculation of the iron minerals using starch, removal of the very fine siliceous gangue by desliming, and flotation of the coarser gangue to produce a high-grade iron oxide concentrate and a froth tailing.

Description of Flowsheet, Equipment, and Test Conditions

Figures 1 and 2 show the equipment used and describe the pulp flows through the selective flocculation and cationic flotation system. The

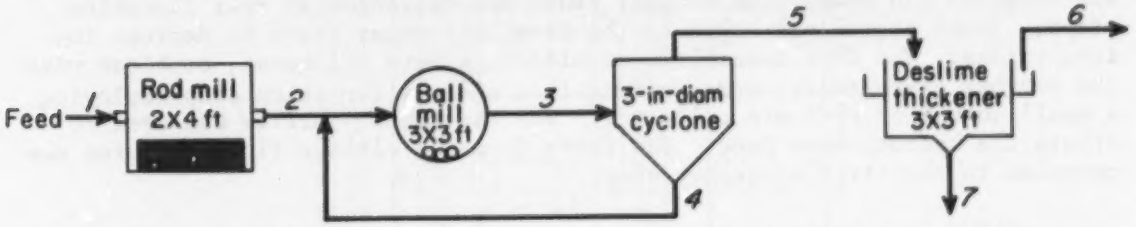

FIGURE 1. - Flowsheet for the grinding, selective flocculation, and desliming processes.

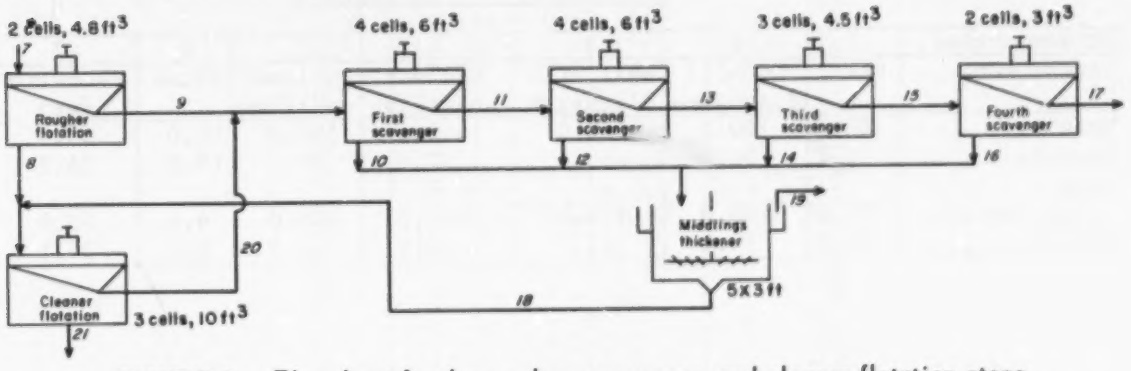

FIGURE 2. - Flowsheet for the rougher, scavenger, and cleaner flotation steps.

metallurgical data derived from the pilot plant operations are shown in tables 3 and 4. Minus 5/8-inch material at 900 lb/hr was diluted with reclaimed water to 64.4 pct solids and fed into a rodmill. The oxidized taconite was ground with the rod charge shown in table 5. Rodmill power requirements were 8.8 kwhr/t, and rod wear was 2.07 lb/t. Sodium silicate and sodium hydroxide were added to the rodmill to disperse the pulp and regulate the pH (table 6). The rodmill discharge containing about 43 pct minus 400 mesh (table 7) was pumped to an overflow-type ball mill. The ball charge shown in table 5 yielded a product that contained about 55 pct minus 400-mesh material. Ball mill power requirements 16.9 kwhr/t, and ball wear was 4.94 lb/t. After the ball mill discharge was diluted to 41 pct solids, it was classified in a hydrocyclone. The cyclone underflow containing about 45 pct minus 400-mesh material was sent back to the ball mill for regrinding. The cyclone overflow, which contained about 91 pct minus 400-mesh material and had a pulp pH of 11.0, was flocculated with causticized pearl starch and deslimed in a thickener. The slime or thickener overflow had a rise rate of 1.7 in/min and was pumped to the water reclamation system. The thickener underflow at about 35 pct solids was pumped to the rougher flotation step where a cationic collector, Arosurf MG-98A[6], which is a mixture of 8- and 10-carbon-chain alkyl oxypropylamine, was added to the pulp. The rougher froth was scavenged in four flotation steps. Pearl starch was added to the first scavenger froth to depress the iron oxides. The four underflows or middlings were thickened, combined with the rougher concentrate, and upgraded in a cleaner flotation step employing a small amount of cationic collector. The thickener overflow was used to dilute the hydrocyclone feed. The froth from the cleaner flotation step was recycled to the first scavenger step.

TABLE 3. - Analysis of components in each stream of the grinding, selective flocculation, and desliming processes shown in figure 1, percent

Stream number.	1	2	3	4	5	6	7
Stream name...	Feed	Rodmill discharge	Ball mill discharge	Cyclone underflow	Cyclone overflow	Slime	Flotation feed
Weight.....	100.0	100.0	543.0	443.0	100.0	12.0	88.0
Iron.....	36.1	36.1	50.4	53.6	36.1	19.8	38.3
Iron distribution.	100.0	100.0	758.0	658.0	100.0	6.6	93.4
Solids.....	94.8	64.4	67.7	68.5	14.8	2.8	35.1

[6]Reference to specific trade names is made for information only and does not imply endorsement by the Bureau of Mines.

TABLE 4. - Analysis of components in the rougher, scavenger, and cleaner flotation streams shown in figure 2, percent

Stream number.....	7	8	9	10	11	12	13
Stream name.....	Flotation feed	Rougher Underflow	Rougher froth	First scavenger		Second scavenger	
				Underflow	Froth	Underflow	Froth
Weight.....	88.0	42.6	45.4	4.1	55.6	7.0	48.6
Iron.....	38.3	60.6	17.4	60.0	21.2	60.1	15.6
Iron distribution.....	93.4	71.5	21.9	6.8	32.7	11.7	21.0
Solids.....	35.1	20.7	20.7	5.8	16.4	3.4	19.1
Stream number.....	14	15	16	17	18	20	21
Stream name.....	Third scavenger		Fourth scavenger		Middlings thickener underflow[1]	Cleaner flotation froth	Concentrate
	Underflow	Froth	Underflow	Froth			
Weight.....	3.5	45.1	5.2	39.9	19.8	14.3	48.1
Iron.....	57.2	12.4	46.2	8.0	55.9	44.4	63.5
Iron distribution.....	5.5	15.5	6.7	8.8	30.7	17.6	84.6
Solids.....	6.0	16.3	3.4	25.6	8.6	9.9	14.7

[1]All components were negligible in the middlings thickener overflow.

TABLE 5. - Rodmill and ball mill media charge

Rodmill				Ball mill		
Rod diameter, inches	Number of rods	Weight		Ball diameter, inches	Weight	
		Lb	Pct		Lb	Pct
1	14	40	3.0	5/8 x 3/4	400	20.0
1-1/2	15	340	22.0	3/4 x 7/8	900	45.0
2	20	800	51.0	7/8 x 1	600	30.0
2-1/2	6	390	24.0	1 x 1-1/4	110	5.0
Total	55	1,570	100.0	NAp	2,010	100.0

NAp Not applicable.

TABLE 6. - Reagent suite

Reagent	Function	Addition point	Amount added, lb/t of feed
SELECTIVE FLOCCULATION AND FLOTATION			
Sodium hydroxide	Dispersant-pH regulator	Rodmill	1.68
Sodium silicate	Dispersant	do	1.72
C-165 pearl starch[1]	Selective flocculant	Conditioner before desliming thickener.	.56
Do[1]	Depressant	First scavenger flotation froth.	.28
Arosurf MG-98A[2]	Collector	Rougher flotation feed.	.235
Do[2]	do	Cleaner flotation feed.	.06
WATER RECLAMATION			
Lime	Coagulant	Slime-tailing thickener.	3.47
Superfloc 330[3]	Flocculant	do	.067
Do[3]	do	Concentrate thickener.	.033

[1]A product of Grain Processing Corp., Muscatine, Iowa.
[2]A product of Ashland Chemical Co., Columbus, Ohio.
[3]A product of American Cyanamid Co., Wayne, N.J.

TABLE 7. - Average screen analyses of grinding circuit products, cumulative weight-percent retained

Sieve size, mesh[1]	Rodmill discharge	Ball mill discharge	Cyclone underflow	Cyclone overflow
Plus 48	2.1	NAp	NAp	NAp
48 x 65	6.5	1.0	1.3	NAp
65 x 100	16.8	2.8	3.4	NAp
100 x 150	28.3	6.2	7.5	1.7
150 x 200	43.2	19.3	22.6	2.4
200 x 270	50.9	32.5	38.8	3.8
270 x 325	52.6	35.7	44.2	7.2
325 x 400	57.4	45.2	55.1	9.1
Minus 400	42.6	54.8	44.9	90.9

NAp Not applicable.
[1]Standard Tyler sieve sizes.

Metallurgical Results

The average metallurgical results (table 8) were obtained over a period of seven consecutive 8-hr pilot plant shifts. Samples for chemical analysis were taken from the pulp streams twice during an 8-hr shift. The first set of three sample cuts was taken 2 hr into the shift and over a 1/2-hr period. The second set of 5 sample cuts was taken 6 hr into the shift and over a 1-hr period. The first sample set was a check on the second set and duplicated the metallurgical data obtained in the second set. The average concentrate analysis is shown in table 9. Concentrates averaging 63.5 pct iron, 4.3 pct silica, and 48.1 wt-pct of the feed with an accompanying iron recovery of 84.6 pct were obtained. The slimes averaging 19.8 pct iron and 12 wt-pct of the feed gave a 6.6 pct iron loss. The tailing averaged 8.0 pct iron and contained 8.8 pct of the iron in the feed.

TABLE 8. - Metallurgical results, pct[1]

Product	Weight	Iron	Silica	Iron distribution
Feed	100.0	36.1	44.5	100.0
Concentrate	48.1	63.5	4.3	84.6
Slime-tailing	51.9	10.7	NAp	15.4
Slime	12.0	19.8	NAp	6.6
Tailing	39.9	8.0	NAp	8.8

NAp Not applicable.
[1]Average data for samples taken over 7 consecutive 8-hr shifts.

TABLE 9. - Concentrate analysis, pct

Fe_2	63.5	CaO	0.46	Na_2O	0.1
Fe	.9	S	.034	C	.18
SiO_2	4.3	P	.037	TiO_2	.27
Al_2O_3	.40	Mn	.17	LOI[1] at 400° C	2.8
MgO	.14	K_2O	.05	LOI[1] at 1,000° C	3.4

[1]LOI = loss on ignition.

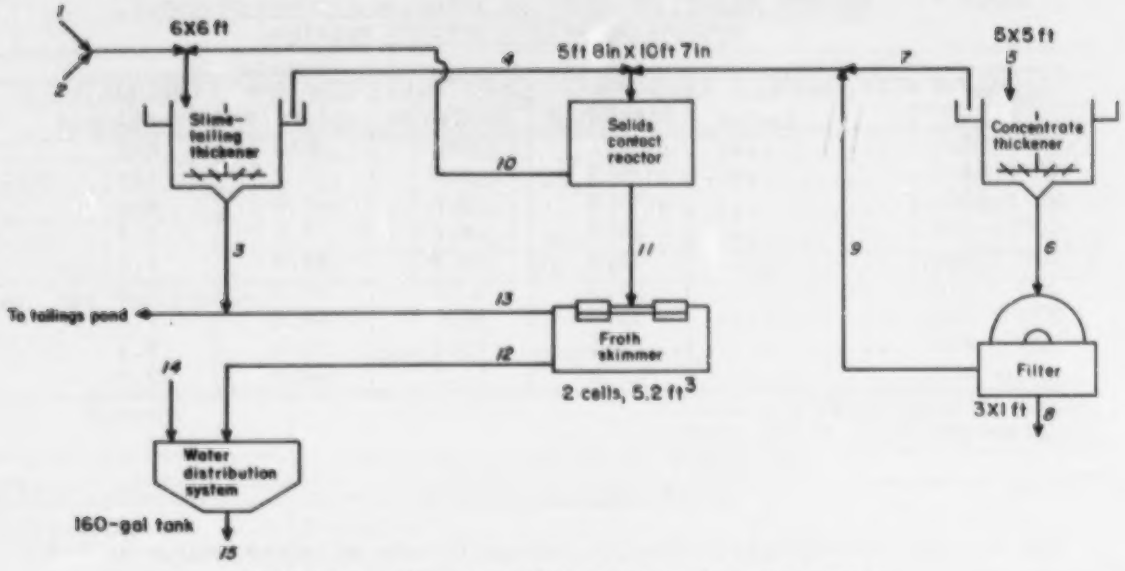

FIGURE 3. - Flowsheet of the water reclamation system.

Water Reclamation

Figure 3 is the water reclamation flowsheet used to treat the pilot plant waste streams. The combined slime and froth tailing at about 9 pct solids was flocculated with 3.47 lb/t of hydrated lime and 0.067 lb/t of Superfloc 330 before being treated in the slime-tailing thickener. The thickener overflow was pumped to a soldis contact reactor (SCR), which is a rectangular compartmented water treatment tank that employs the principles of sludge-blanket clarification. The thickener underflow controlled at 50 pct solids to prevent excess water loss, was pumped to a tailing pond. The SCR underflow was recycled to the slime-tailing thickener, and the SCR overflow was sent to the froth skimmer. Excess froth was removed by the skimmer and was also routed to the tailing pond. The froth skimmer underflow was pumped to the water distribution system, and makeup water was added to satisfy plant water requirements.

The concentrate was fed to a thickener and flocculated with 0.033 lb/t of cationic flocculant Superfloc 330. Concentrate thickener underflow was adjusted to 60 pct solids and filtered on a rotary drum filter. Filter cakes having an average moisture content of 13 pct and a Blaine surface area of 2,850 cm^2/g were obtained. The filtrate was combined with the concentrate thickener overflow and routed to the SCR. An average of 88 pct of the total water used or 17.7 gal/min was recovered and recycled. Water losses totaled 2.4 gal/min. Additional water would be available for plant use if tailing pond water were recycled. The reclaimed water had an average pH of 11, turbidity of 480 ppm silica equivalent and calcium ion content of 7.6 ppm. The material balance for the water reclamation system is shown in table 10.

TABLE 10. - Material balance for the water reclamation flowsheet shown in figure 3

Stream number.....	1	2	3	4	5	6	7	
Stream name.....	Slime	Tailing	Slime-tailing thickener underflow	Slime-tailing thickener overflow	Concentrate	Concentrate thickener underflow	Concentrate thickener overflow	
Solids.....pct..	2.8	25.6	50.0	0.03	14.7	60.0	0.04	
Pulp.....gal/min..	11.3	3.6	1.6	14.2	5.2	0.8	4.4	
Stream number.....	8	9	10	11	12	13	14	15
Stream name.....	Filter cake	Filtrate	Solids contact reactor underflow	Solids contact reactor overflow	Froth skimmer underflow	Froth skimmer froth	Make-up water	Water to plant
Solids.....pct..	87.0	Neg[1]	10.3	Neg[1]	Neg[1]	Neg[1]	Neg[1]	Neg[1]
Pulp.....gal/min..	0.3	0.5	0.9	18.2	17.7	0.5	2.4	20.1

[1]Neg = negligible.

Table 11 shows the average amine and sodium analyses for the froth skimmer overflow and underflow, slime-tailing thickener overflow, and the combined makeup and recycled water used in the plant. About 93 pct of the amine in the reclaimed water was removed in the froth skimmer. Sodium in the recycled water remained relatively constant throughout the tests.

TABLE 11. - Averaged sodium and amine analyses of water reclamation streams, ppm

Stream	Amine	Sodium
Slime-tailing thickener overflow	0.7	109
Froth skimmer overflow	24.2	104
Froth skimmer underflow	.7	109
Water to plant	.6	96

SUMMARY AND CONCLUSIONS

The metallurgical results from the application of the selective flocculation and flotation process to the second of three bulk samples of western Mesabi oxidized taconite were presented. The bulk sample, representing about 1/2 to 1 billion tons of material and containing 36.1 pct iron was upgraded to a concentrate grade of 63.5 pct iron and 4.3 pct silica. Iron recovery was 84.6 pct. The first bulk sample, representing about 2 billion tons of material and containing 35.3 pct iron, yielded concentrates containing 61.7 pct iron and 5.1 pct silica. Iron recovery was 67.2 pct. The major difference between the samples was the hematite-to-goethite ratio, which was 1.1 to 1 for the first sample and 3 to 1 for the sample reported in this paper. Liberation of the iron oxides from the gangue minerals occurred at 400 mesh (37 μm) for the first sample and 325 mesh (44 μm) for the second. The sample discussed in this paper responded more favorably to flotation than the first sample and required one-half as much collector (0.28 lb/t compared to 0.56 lb/t) to produce acceptable concentrates. Blaine surface areas of the concentrates were similar and averaged about 2,850 cm^2/g. These results showed that commercially acceptable concentrates can be made from oxidized taconites containing appreciable amounts of the hydrous iron oxide, goethite.

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